NASA CR OF	SSION NUMBER) (THRU) (PAGES) (CODE) (CATEGORY)
B	GPO PRICE \$ OTS PRICE(S) \$
Caronal SHED PRELIMINAN	Microfiche (MF)

BAUSCH & LOMB INCORPORATED

ROCHESTER 2, NEW YORK

Grating Groove Formation in Au and Au-Ge Alloys

NONR-4277 (00) (X) \mathcal{R}

NONR-4277 (00) (X)

Final Report of Work in 1964

Submitted by Bausch & Lomb Incorporated March 26, 1965

> C. Frank Mooney **Grating Research Section**

Grating Groove Formation in Au and Au-Ge Alloys NONR-4277 (00)(X)

Final Report of Work Performed by Bausch & Lomb in 1964

Spectroscopic and Reflectance Measurements were Made Using Naval Research Laboratory
Equipment

Submitted by

Bausch & Lomb Incorporated

March 26, 1965

C. Frank Mooney
Grating Research Section

CONTENTS

Α.	SUMMARY OF	CONCLUSIONS	FROM 1964	CONTRACT	WORK	2
B.	TEST GRATII	NG SUMMARY S	HEETS			4

- A. SUMMARY OF CONCLUSIONS FROM 1964 CONTRACT
- 1. Gold films made under some conditions can be burnished with smooth grating grooves of some types.
 - a. Gold deposited rapidly in good vacuum is too malleable to rule well.
 - b. Gold that is somewhat less malleable can be ruled without oil.
 - c. Gold that is least malleable can be ruled with 3 degree to 7 degree blaze angles conventionally under oil.
 - d. Gold-germanium alloy films are, in contrast to pure gold, useful for grooves with a steep blaze face.
- 2. The electron microscope reliably estimates groove "depth" of a replicated specimen if the groove surface of the original grating is smooth and if there is little change of groove shape from one area of the grating to another. An accurate specimen replica method has been discovered and used for all of the test gratings ruled in this contract. At the very end of the experimental work done in this contract, a 2400 groove per millimeter grating with a 30 nm nominal blaze was attempted entirely under electron microscope control. The slowness of the operation combined in this case with an error of procedure to obscure the success of the project.
- 3. Vacuum deposition variables and lumpy conditions on the films are a main source of difficulty with plano gratings in gold.

Many of these difficulties can be avoided by using transfer films; i.e., films where the exposed surface was originally in contact with smooth polished glass. Deformation of the bonding cement seems then to limit the results.

- 4. Cumulative diffractance was highest generally for gratings with sharp, flat grooves that have clean groove edges.

 Generally, only the smooth grooved gratings had significant flux in any orders except 0, 1, and 2.
- 5. Most other observations are mutually contradictory. It would seem that further control of important variables is essential for detailed progress.

B. TEST GRATING SUMMARY SHEETS

Forty of the overnight test gratings made in this contract were studied in sufficient detail to warrant the preparation of summary sheets. The information is necessarily condensed and several of the notations may need to be explained.

In the summary of observations, conditions observed during filming and in ruling setup are reported. Where the same film was used or where the same deposition run was used for more than one test ruling area, the other ruling number is included.

In the groove profile, the reported measurements were made from an electron micrograph. The groove angle is not drawn to scale because the ordinate and abscissa are on different scales. The sequence of ruling the grooves is shown by an arrow. Several of the grating micrographs were not measured.

A shorthand notation on the cumulative diffractance needs to be explained:

$$\sum 1 = 0 + 1$$
 orders combined,

$$\sum 2 = 0 + 1 + 2$$
 orders combined,

$$\sum 3 = 0 + 1 + 2 + 3$$
 orders combined.

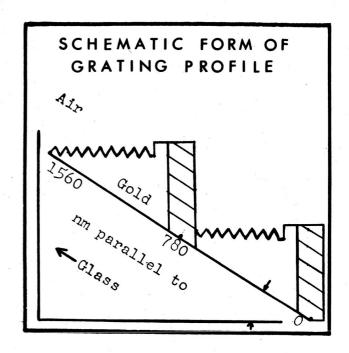
In this form of presentation, crossing curves are avoided. The spectrum scale is linear in wavenumber, but for personal comfort

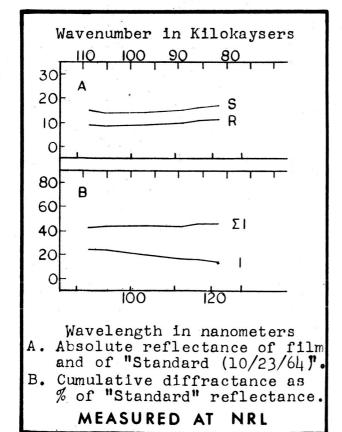
the abscissa is plotted with wavelength increasing from left to right.

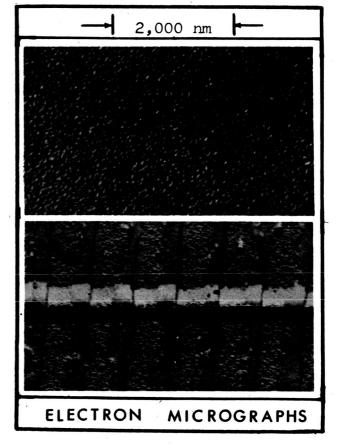
Both micrographs are to the same scale. The top line of the shadow corresponds to the profile of the grating with air above and metal below but with the vertical scale exaggerated about three times.

OBSERVATIONS

Substrate - Shined glass plate
Deposition - 32 microtorr
Electron gun, C crucible
Au: 270 nm: .47 nm/sec
Ruling - 1280/mm
Steep burr, resurgent
Au adhesion to tool locally
Tool bounced, large nuggets
An unruled diagonal band
Burnishing - .3 complete
Groove edge wave - .3 groove

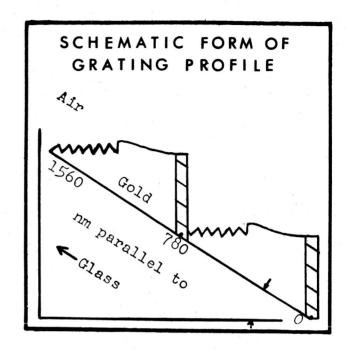


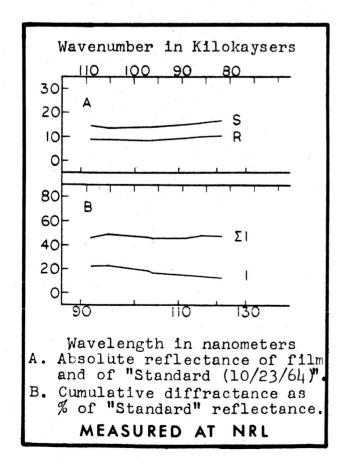


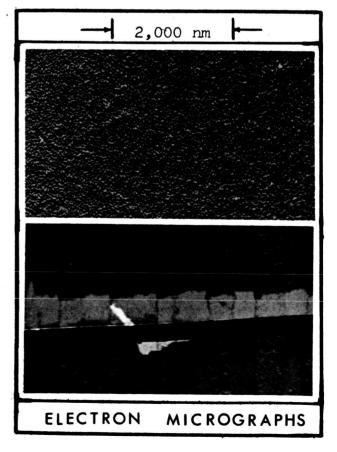


OBSERVATIONS

Substrate - Shined glass plate
Deposition - 150 microtorr
Electron gun, C crucible
Au: 130 nm: .36 nm/sec
Ruling - 1280/mm
Slight burr step
Grooves difficult to form,
then poorly shaped
Tool bounce and stripes
Burnishing - .5 complete
Groove edge wave - .05 groove.

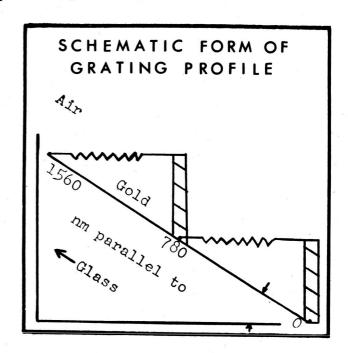


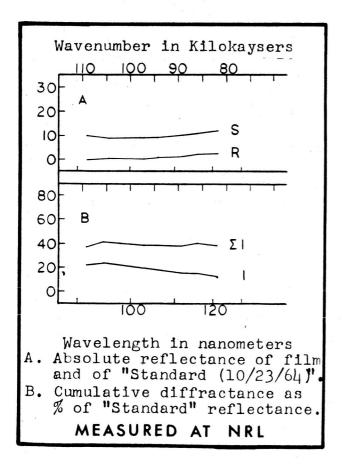


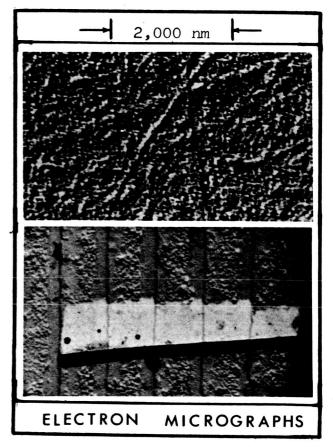


OBSERVATIONS

Substrate - Shined plate
Deposition - 120 microtorr
Electron gun, C crucible
Au: 270 nm: .9 nm/sec
Ruling - 1280/mm
.4 fringe burr
Some tool bounce on nuggets
and streaks
Grooves have good shape
Burnishing - .6 complete
Groove edge wave - .1 groove.

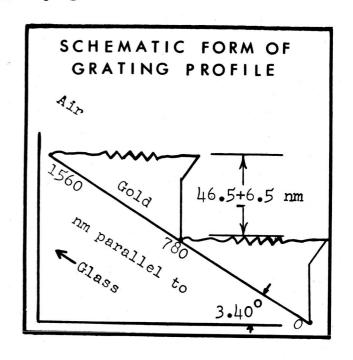


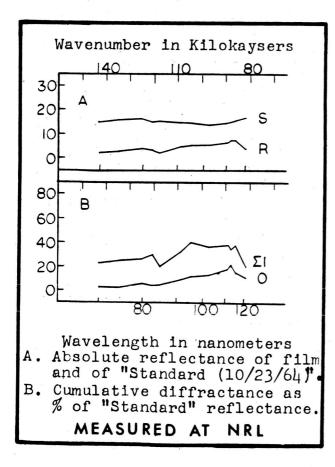


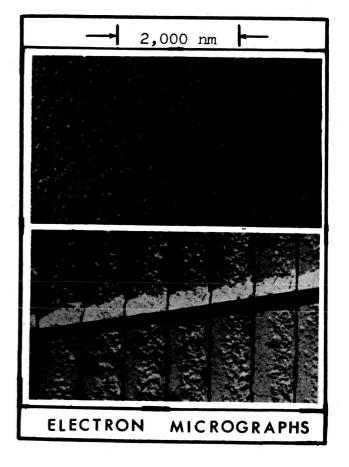


OBSERVATIONS

Substrate - Shined glass plate
Deposition - 135 microtorr
Electron gun, C crucible
Au: 620 nm: .63 nm/sec
Ruling - 1280/mm
.3 fringe burr
Ruling bothered by nuggets
and tool bounce
Rough groove profile
Burnishing - .4 complete
Groove edge wave - .2 groove







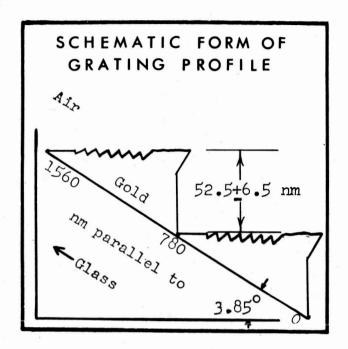
OBSERVATIONS

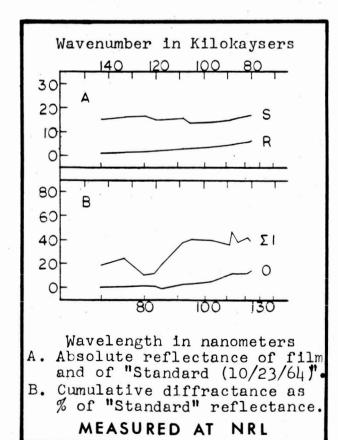
Substrate - Shined glass plate
Deposition - 50 microtorr
Electron gun, C crucible
Au: 760 nm: 6.3 nm/sec
Ruling - 1280/mm
.3 fringe burr
Excessive interaction of
tool with nuggets

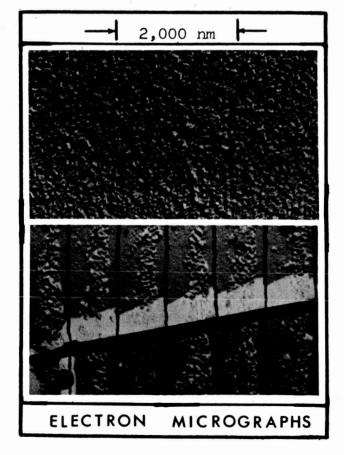
Burnished groove areas smooth

Burnishing - .4 complete

Groove edge wave - .2 groove







OBSERVATIONS

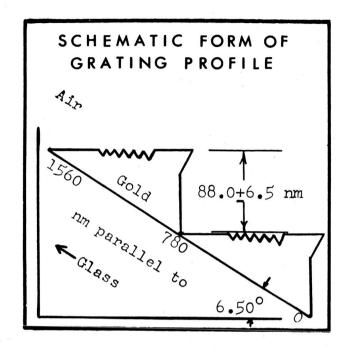
Substrate - Shined glass plate
Deposition - 120 microtorr
Electron gun, C crucible
Au: 400 nm: .34 nm/sec
Ruling - 1280/mm

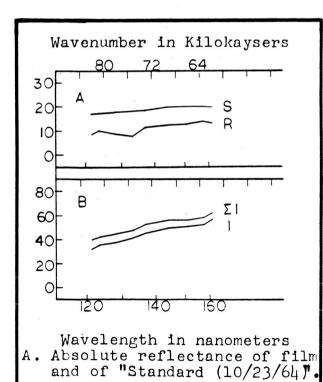
.7 fringe burr, resurgent Ruled well despite burr Small jumps and streaks

Heavy streaks in one section

Burnishing - .7 complete

Groove edge wave - .2 groove





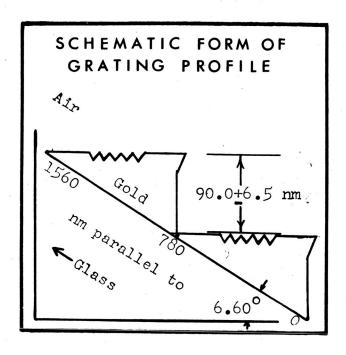
B. Cumulative diffractance as % of "Standard" reflectance.

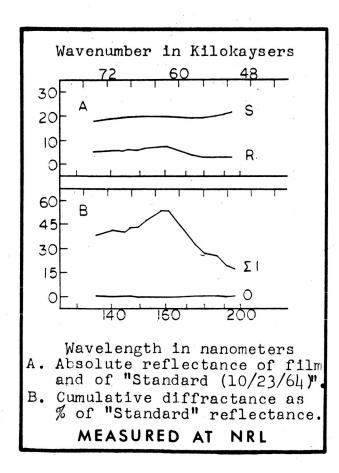
MEASURED AT NRL

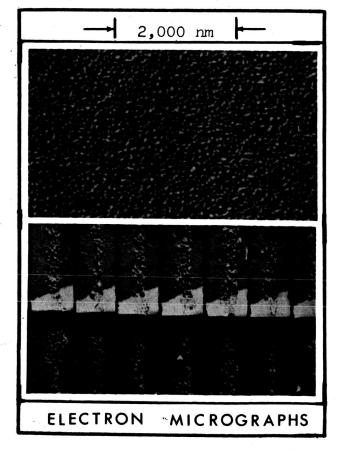
ELECTRON MICROGRAPHS

OBSERVATIONS

Substrate - Shined glass plate
Deposition - 55 microtorr
Electron gun, C crucible
Au: 1100 nm: 9.2 nm/sec
Ruling - 1280/mm
.6 fringe burr
Ruled well, grooves smooth
Ruling streaks everywhere
Center of ruling poorer
Burnishing - .7 complete
Groove edge wave - .15 groove







OBSERVATIONS

Substrate - Shined glass plate
Deposition - 60 microtorr
Electron gun, C crucible
Au: 1650 nm: 12 nm/sec

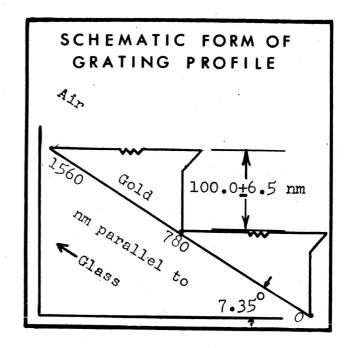
Ruling - 1280/mm

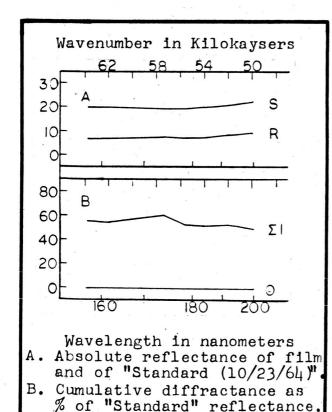
•7 fringe burr
Ruled smoothly with streaks

and jumps at nuggets Good appearance

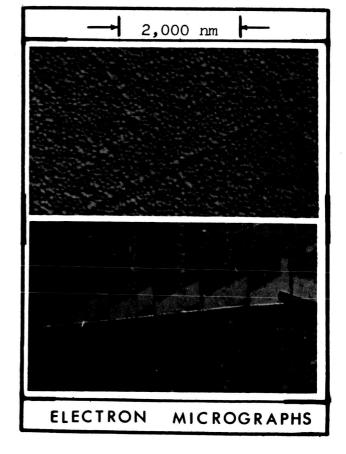
Burnishing - .8 complete

Groove edge wave - .3 groove





MEASURED AT NRL



OBSERVATIONS

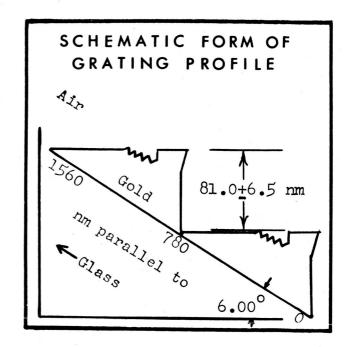
Substrate - Shined glass plate
Deposition - 40 microtorr
Electron gun, C crucible
Au: 1090 nm: 13.5 nm/sec

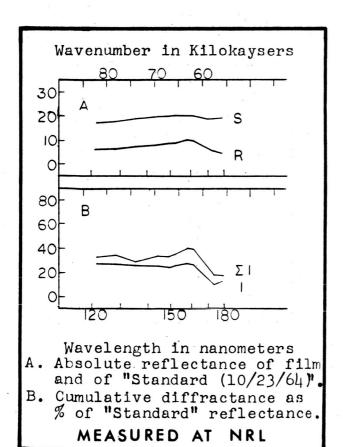
Ruling - 1280/mm

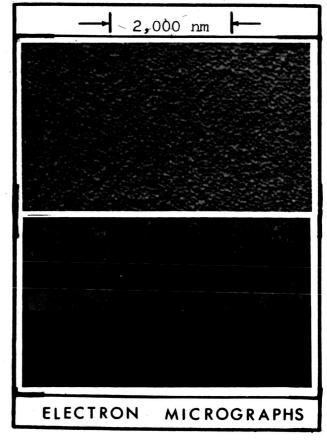
.8 fringe burr

Ruled poorly, much jumping
and prominent streaks

Large resurgent burr
Burnishing - .6 complete
Groove edge wave - .1 groove







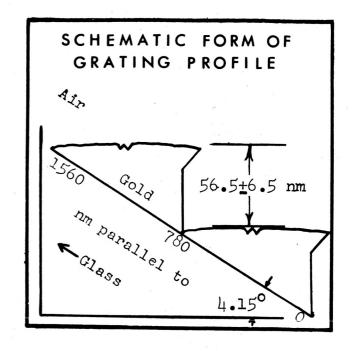
OBSERVATIONS

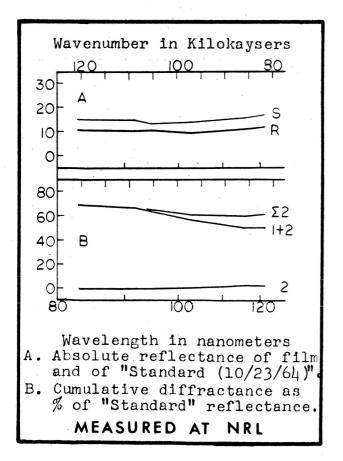
Substrate - Shined glass plate
Deposition Joule heated source
Au: very thin: very fast
Ruling - 1280/mm, Dry
High burr
Could not rule with oil,
Grooves improved toward end

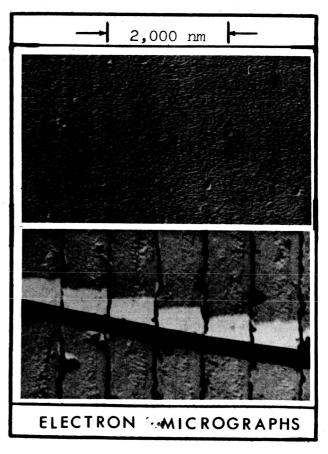
Best groove form to that time

Groove edge wave - .25 groove

Burnishing - .9 complete







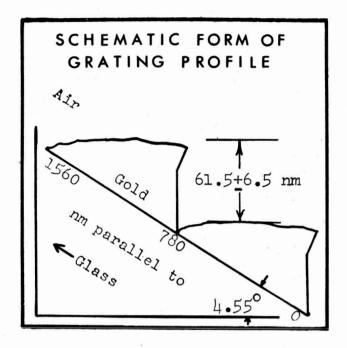
OBSERVATIONS

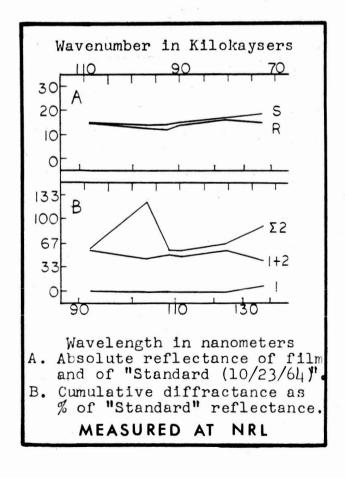
Substrate - Transfer film
Deposition - 40 microtorr
Electron gun, C crucible
Au: 320 nm: 5.3 nm/sec

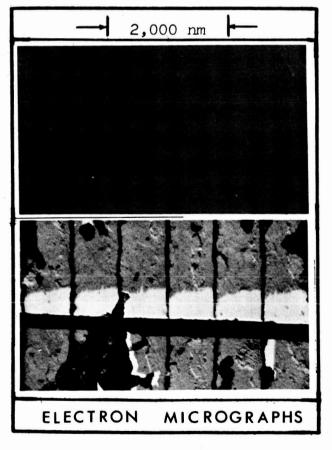
Ruling - 1280/mm

.3 fringe burr Au adhesion to tool caused occasional streaks

Particularly free of nuggets
Burnishing - .8 complete
Groove edge wave - .1 groove



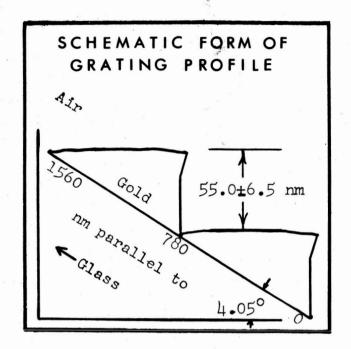


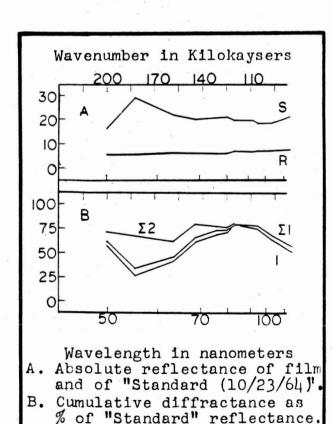


OBSERVATIONS

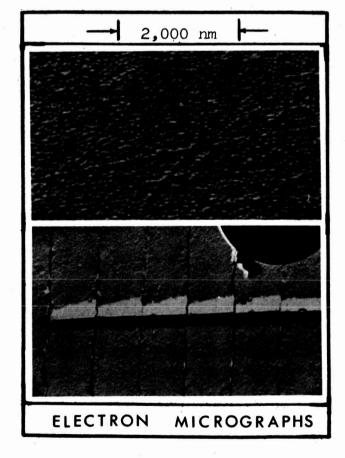
Substrate - Shined glass plate
Deposition - 50 microtorr
Electron gun, C crucible
Au: 520 nm: 5.5 nm/sec
Ruling - 1280/mm, dry
.5 fringe burr
Some Au adhesion to tool,
Ruled well
Nugget clusters caused jumps
Burnishing - complete

Groove edge wave - 15 groove





MEASURED AT NRL



OBSERVATIONS

Substrate - Transfer film

Deposition - 90 microtorr

Electron gun, C crucible

Au: 680 nm: 7.5 nm/sec

Ruling - 1280/mm

.4 fringe burr

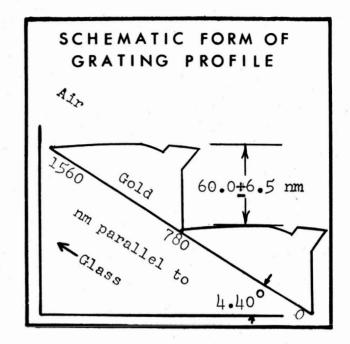
Ruled particularly well from

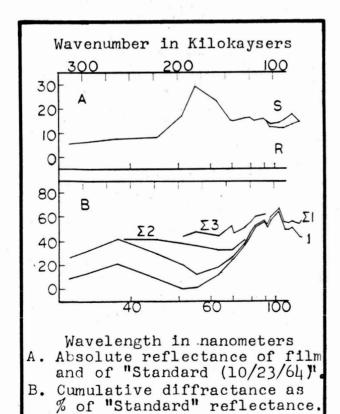
Exceptionally good film

Burnishing - .9 complete

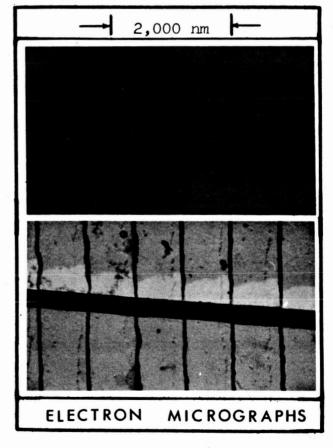
Groove edge wave - .1 groove

start to finish

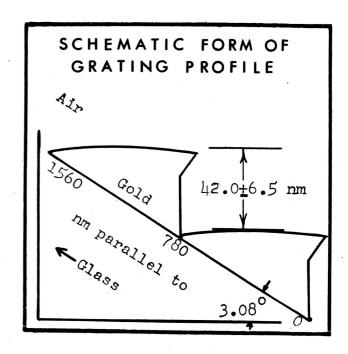


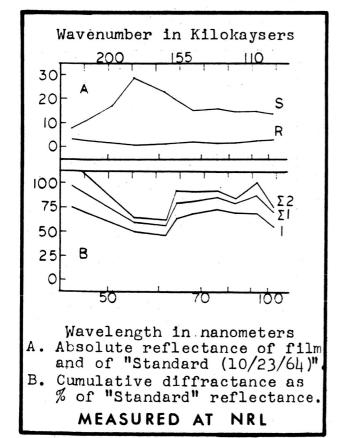


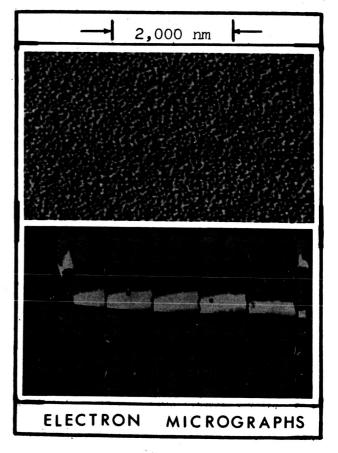
MEASURED AT NRL



OBSERVATIONS
Substrate - Shined glass plate
Deposition - 75 microtorr
Electron gun, C crucible
Au: 1100 nm: 9.2 nm/sec
Ruling- 1280/mm, dry
.4 fringe burr
Streaking, tool disturbed
by nuggets
Exceptionally bright finish
Burnishing - complete
Groove edge wave - .2 groove







OBSERVATIONS

Substrate - Transfer film

Deposition - 100 microtorr

Electron gun, C crucible

Au: 820 nm: 14 nm /sec

Ruling - 1280/mm, dry

.3 fringe burr

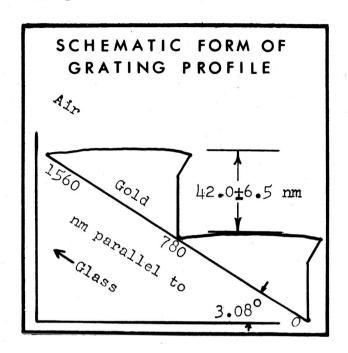
Ruled well

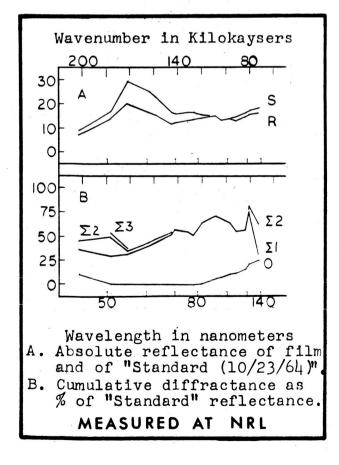
No streaks

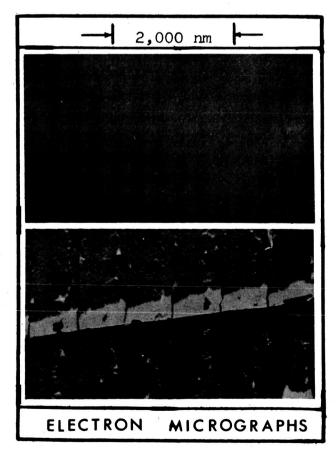
Some traced burr remains

Burnishing - Complete, burr

Groove edge wave - .2 groove

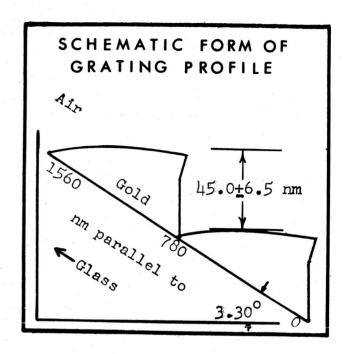


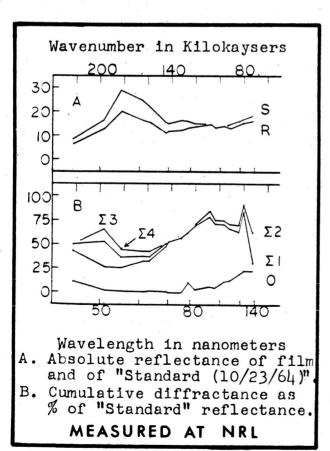


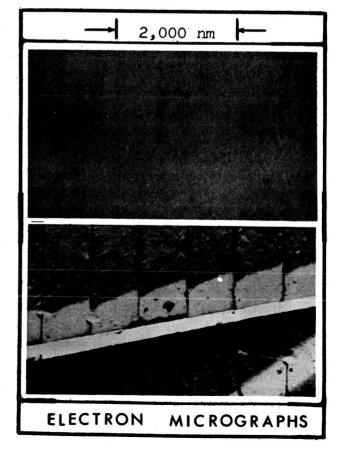


OBSERVATIONS

Substrate - (=25) Transfer film
Deposition - 100 microtorr
Electron gun, C crucible
Au: 820 nm: 14 nm/sec
Ruling - 1280/mm, oil
.2 fringe burr
Numerous streaks appeared
Ruled poorly
Duller than 25 but less burr
Burnishing - Complete
Groove edge wave - .1 groove

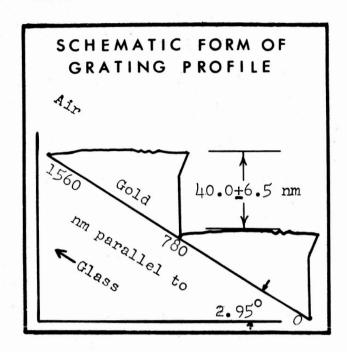


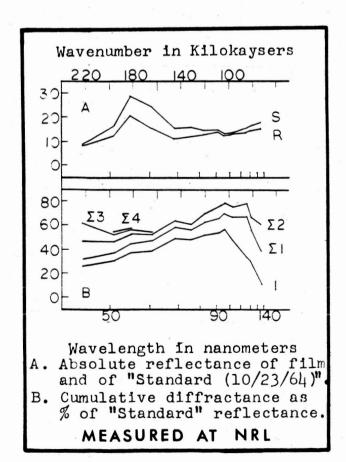


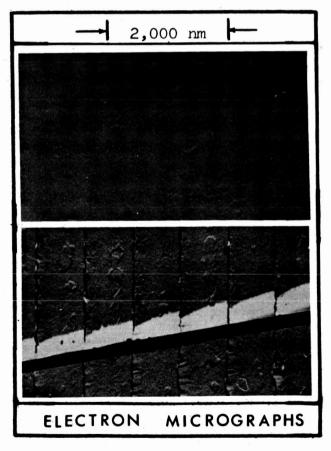


OBSERVATIONS

Substrate- (=29) Transfer film
Deposition - 60 microtorr
Electron gun, C crucible
Au: 380 nm: 6.3 nm/sec
Ruling - 1280/mm, dry
.3 fringe burr
Flat groove face not clear,
some streaks
Good appearance, Mesa texture
Burnishing - Complete
Groove edge wave - .1 groove







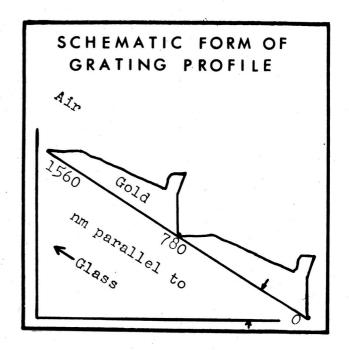
OBSERVATIONS

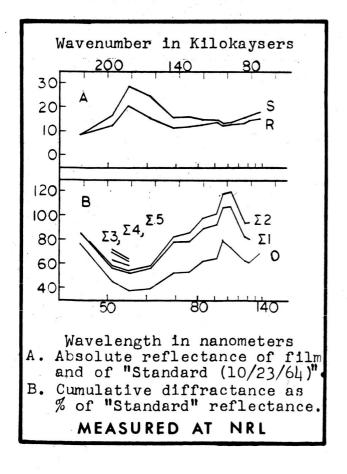
Substrate - (=28) Transfer film
Deposition - 60 microtorr
Electron gun, C crucible
Au: 380 nm: 6.3 nm/sec
Ruling - 1280/mm, oil
.4 fringe burr
Ruled area inferior to test

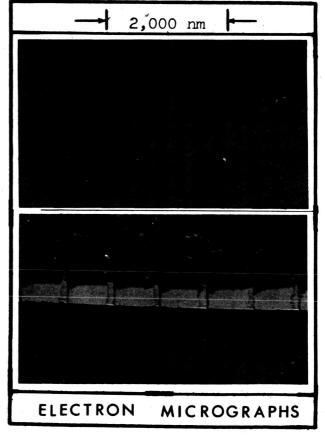
NRL 28 appeared better

Burnishing - Complete, burr Groove edge wave - .05 groove.

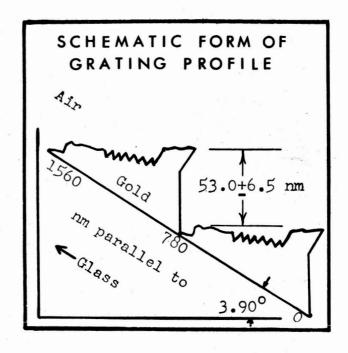
lines, more streaks

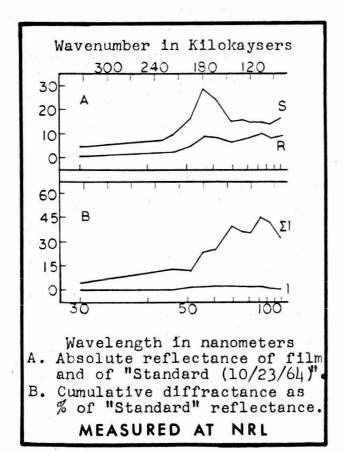


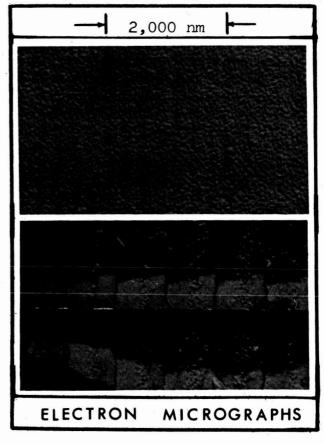




OBSERVATIONS
Substrate - Polished glass
Deposition - With NRL 31
40 microtorr
Electron gun, C crucible
Au: 250 nm: 2.1 nm/sec
Ruling - 1280/mm
.4 fringe burr
Ruled well but some
streaky sections
Not a good ruling
Burnishing - variable, burr
Groove edge wave - .3 groove

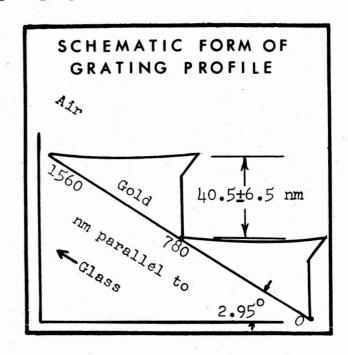


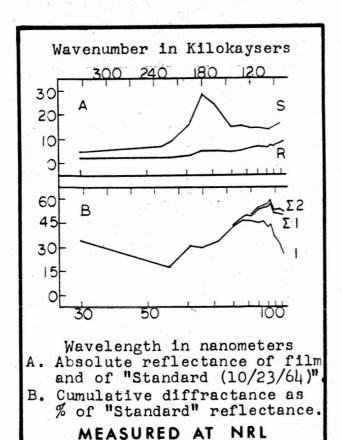


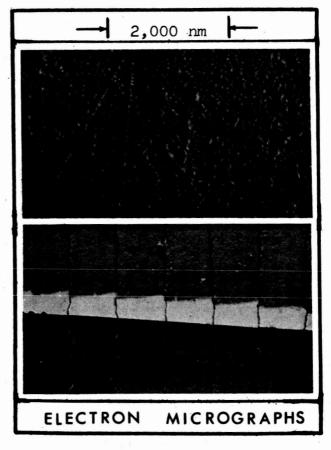


OBSERVATIONS

Substrate - Shined glass plate
Deposition - With NRL 30
40 microtorr
Electron gun, C crucible
Au: 250 nm: 2.1 nm/sec
Ruling - 1280/mm
.4 fringe burr
Did not rule well due
to nuggets
Good locally
Burnishing - Complete
Groove edge wave - .3 groove







OBSERVATIONS

Substrate - Polished glass

Deposition - 35 microtorr

Electron gun, C crucible

Au₈₈ Ge₁₂: 270 nm: 2.6 nm/sec

Ruling - 1280/mm

. fringe burr

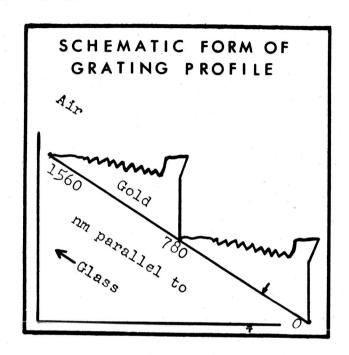
Film not easily burnished,

jumps over nuggets

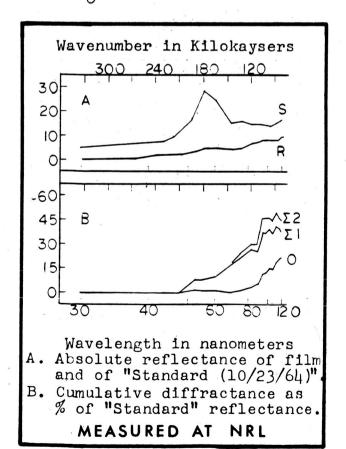
Ruled well but not thoroughly

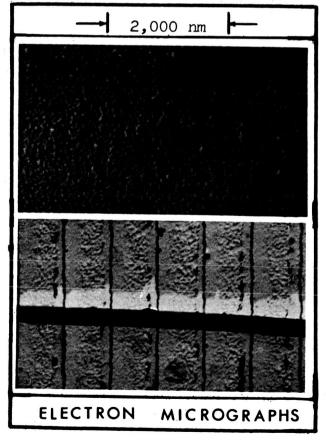
Burnishing - .2 Complete

Groove edge wave - .05 groove



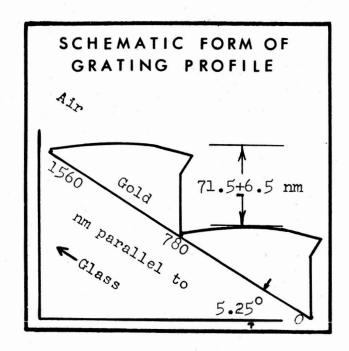
0

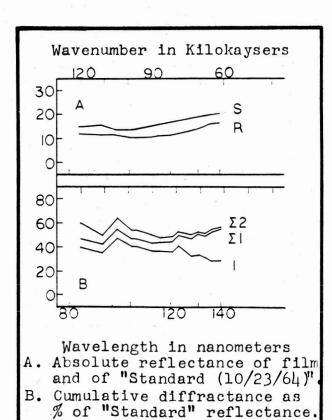




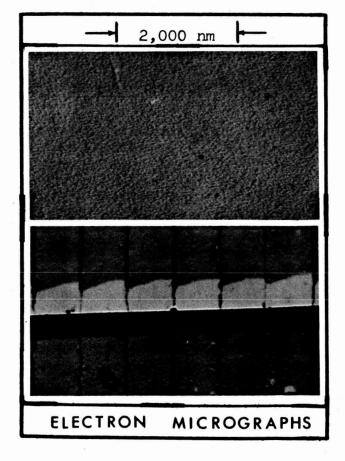
OBSERVATIONS

Substrate - (=34) Transfer film
Deposition - 35 microtorr
Electron gun, C crucible
Au: 140 nm: .ll nm/sec
Ruling - 1280/mm, dry
.2 fringe burr
Ruled well, disturbed cement
not full depth
Blank shifted near end
Burnishing - .5 Complete
Groove edge wave - .15 groove



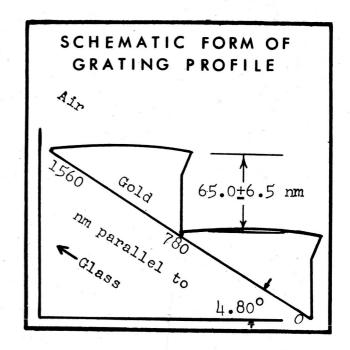


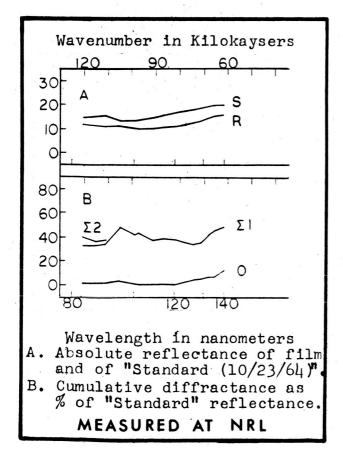
MEASURED AT NRL

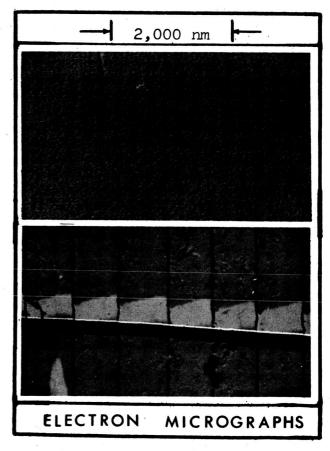


OBSERVATIONS

Substrate - (=33) Transfer film
Deposition - 35 microtorr
Electron gun, C crucible
Au: 140 nm: .ll nm/sec
Ruling - 1280/mm, oil
.3 fringe burr
Ruled well,
Disturbed cement
Excellent ruling
Burnishing - Complete
Groove edge wave - .l groove







OBSERVATIONS

Substrate - (=36) Polished glass

Deposition - With NRL 37,38

50 microtorr

Electron gun, C crucible

Au: 460 nm: 5 nm/sec

Ruling - 1280/mm

3 fringe burr

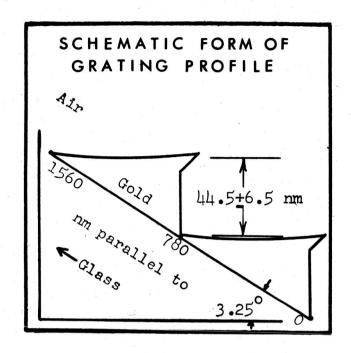
Ruled well except for some

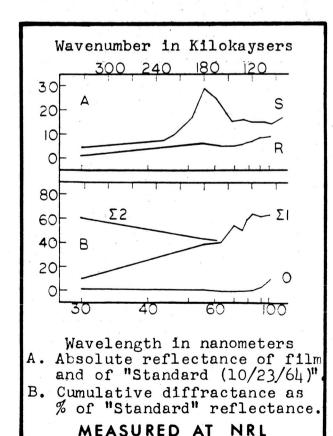
nugget movement

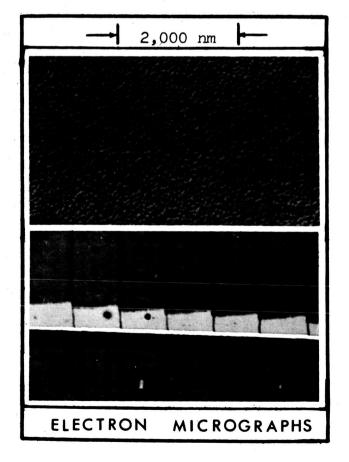
Good ruling

Burnishing - Complete

Groove edge wave - 2 groove

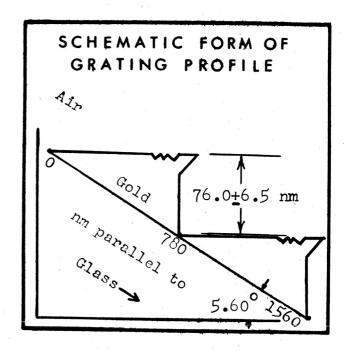


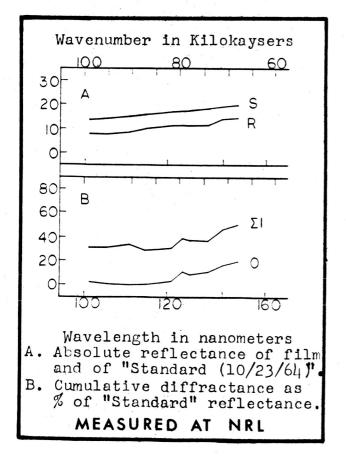


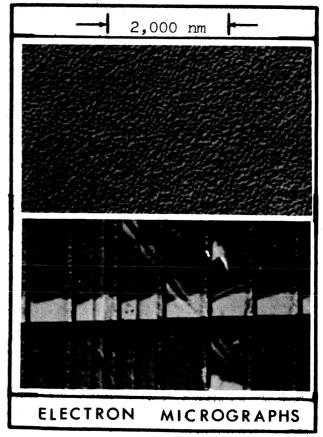


OBSERVATIONS

Substrate-(=35) Polished glass
Deposition - with NRL 37, 38
50 microtorr
Electron gun, C crucible
Au: 460 nm: 5 nm/sec
Ruling - 1280/mm,
.2 fringe burr
Many jumps and streaks
did not rule well
Groove bottom evident
Burnishing - .9 Complete
Groove edge wave - .05 groove







OBSERVATIONS

Substrate - (=38)Polished glass
Deposition - with NRL 35, 36
50 microtorr
Electron gun, C crucible
Au: 460 nm: 5 nm/sec

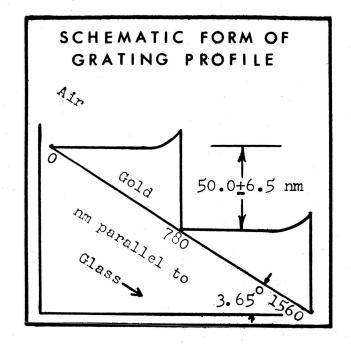
Ruling - 1280/mm

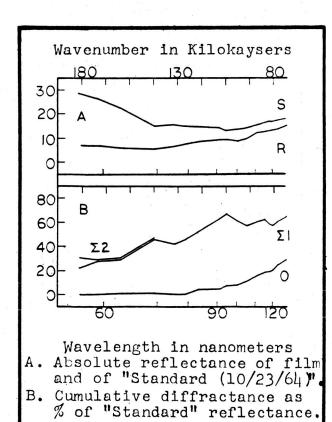
.2 fringe burr

Some areas ruled very well

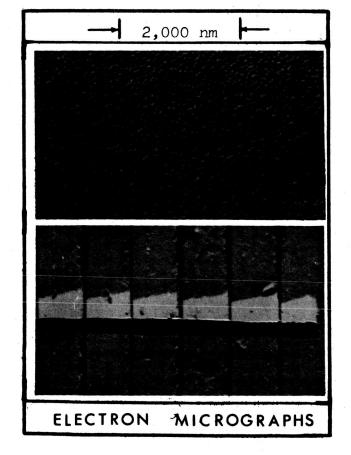
Some not well

Heavier jump marks and streaks
Burnishing - .9 complete
Groove edge wave - none





MEASURED AT NRL

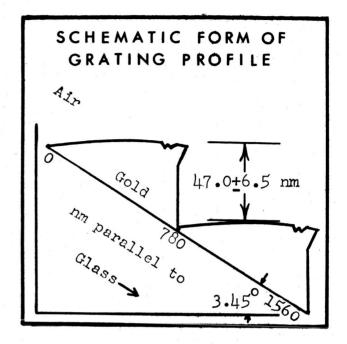


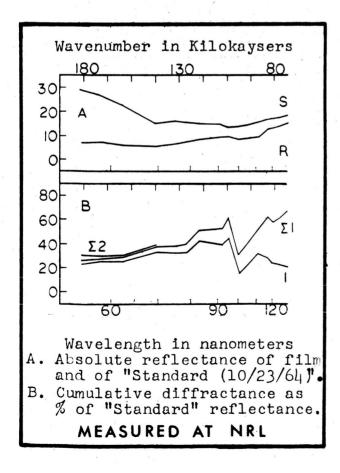
OBSERVATIONS

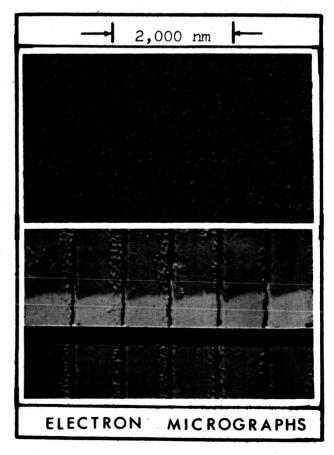
Substrate - (=37) Polished glass
Deposition - with NRL 35, 36
50 microtorr
Electron gun, C crucible
Au: 460 nm: 5 nm/sec

Ruling - 1280/mm,
3 fringe burr
Jumps at nuggets,
Ruled fairly well
Not as bright as 37
Burnishing - .8 complete

Groove edge wave - .1 groove

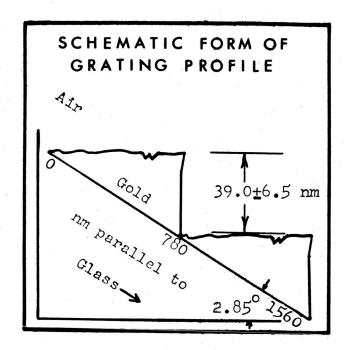


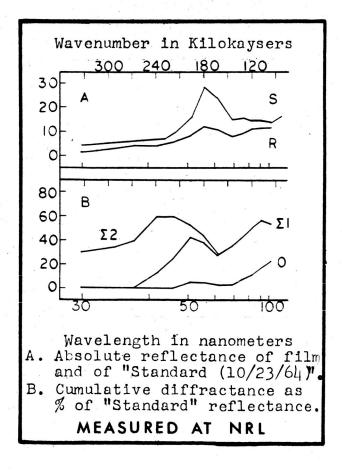


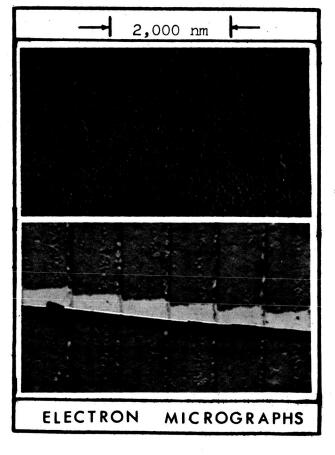


OBSERVATIONS

Substrate - (=40) Polished glass
Deposition - with NRL 41, 42
50 microtorr
Electron gun, C crucible
Au: 250 nm: 5.5 nm/sec
Ruling - 1280/mm,
3 fringe burr
Double ruling may test
engine quality mainly
Similar to 3%, rarely good
Burnishing - .8 complete
Groove edge wave - .05 groove







OBSERVATIONS

Substrate - (=39) Polished glass

Deposition - with NRL 41, 42

50 microtorr

Electron gun, C crucible

Au: 250 nm: 5.5 nm/sec

Ruling - 1280/mm

.4 fringe burr

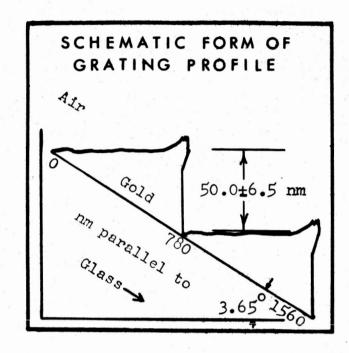
Many nuggets, jumps,

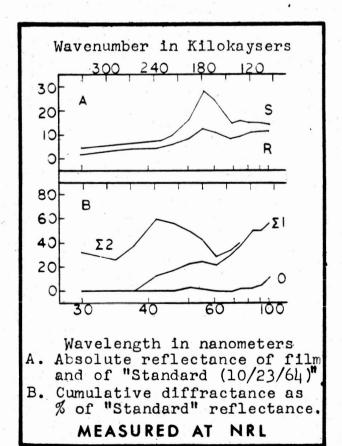
and streaks

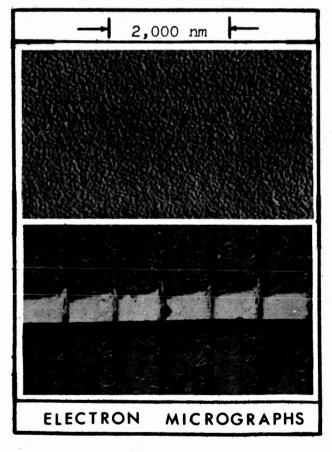
Better than 39

Burnishing - .9 complete

Groove edge wave - .05 groove

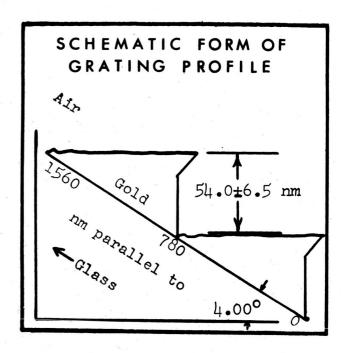


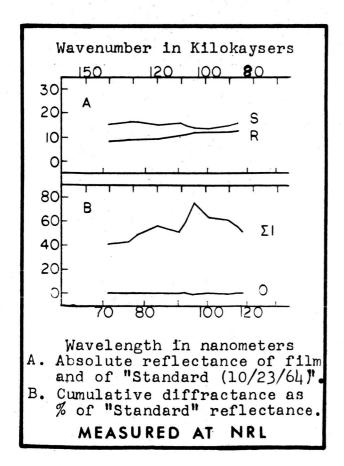


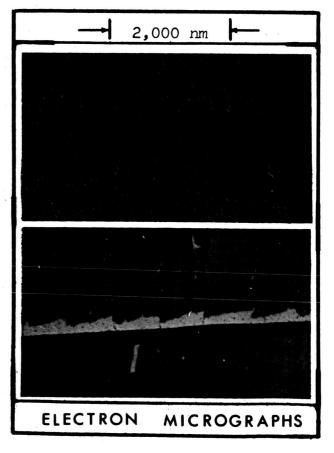


OBSERVATIONS

Substrate- (=42) Polished glass
Deposition - With NRL 39,40
50 microtorr
Electron gun, C crucible
Au: 250 nm: 5.5 nm/sec
Ruling - 1280/mm,
3 fringe burr
Test lines better than
single, ruling
Did not rule well, streaks
Burnishing - Complete
Groove edge wave - .3 groove



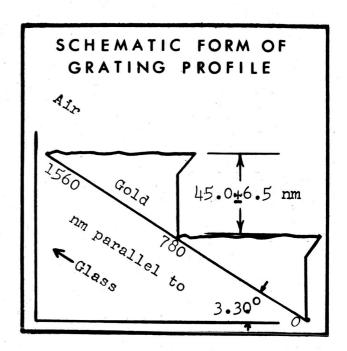


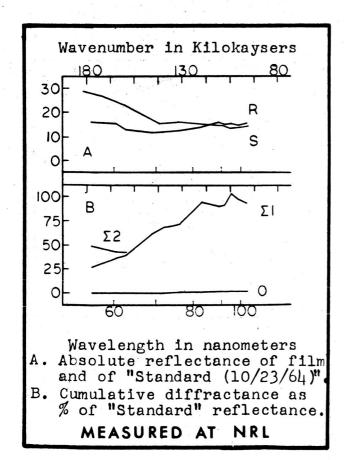


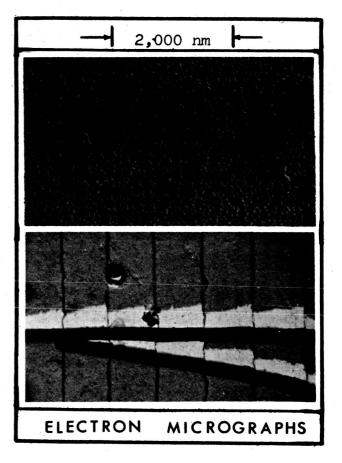
OBSERVATIONS

Substrate-(=41) Polished glass
Deposition - With NRL 39,40
50 microtorr
Electron gun, C crucible
Au: 250 nm: 5.5 nm/sec
Ruling - 1280/mm
.3 fringe burr
Few ruling streaks, fringes
less distinct
Groove shape the same as 41
Burnishing - .9 complete

Groove edge wave - .25 groove







OBSERVATIONS

Substrate - Polished plate

Deposition - With NRL 44

40 microtorr
Electron gun, C crucible
Au: 320 nm: 3.5 nm/sec

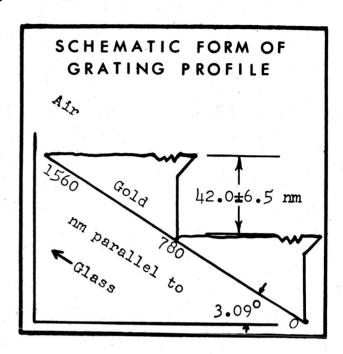
Ruling - 1280/mm

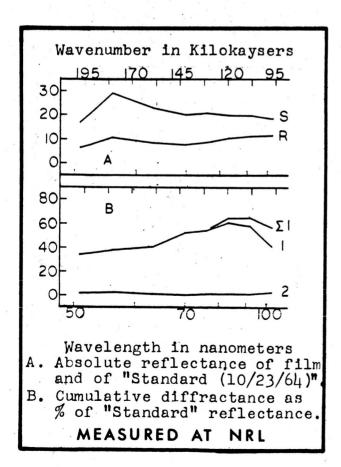
.2 fringe burr
some gold adhesion,
ruled well, some streaks

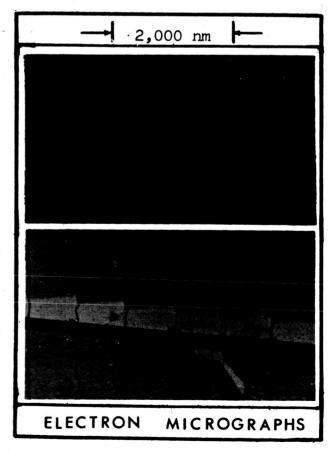
Fringes from grooves indistinct

Burnishing - .9 complete

Groove edge wave - .2 groove



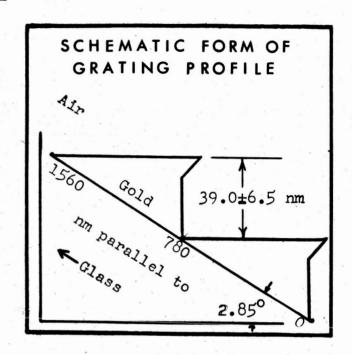


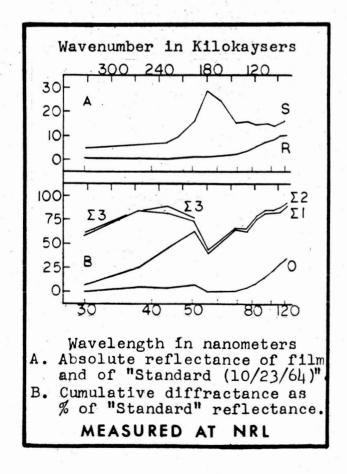


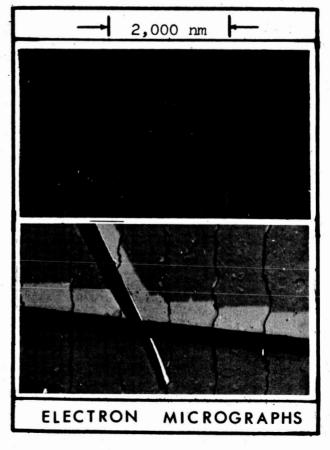
OBSERVATIONS

Substrate - Shined glass plate
Deposition - With NRL 43
40 microtorr
Electron gun, C crucible
Au: 320 nm: 3.5 nm/sec
Ruling - 1280/mm
.2 fringe burr
Some gold adhesion,
ruled well
Much groove width variation
Burnishing - Complete

Groove edge wave - .3 groove







OBSERVATIONS

Substrate - Polished glass

Deposition - 35 microtorr

Electron gun, C crucible

Au₉₄ Ge6: 300 nm: 1.8 nm/sec

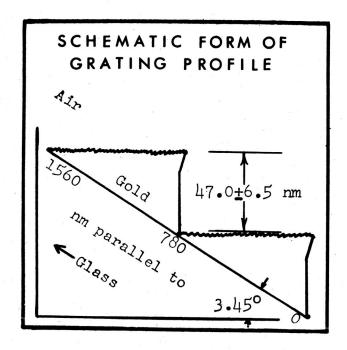
Ruling - 1280/mm

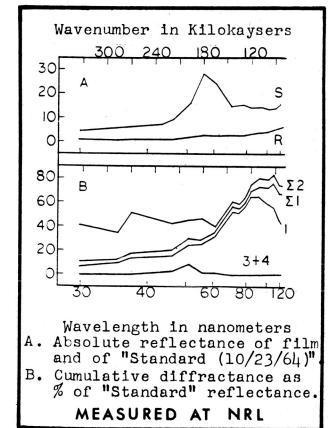
.3 fringe burr
Nuggets disturbed diamond
Some streaks

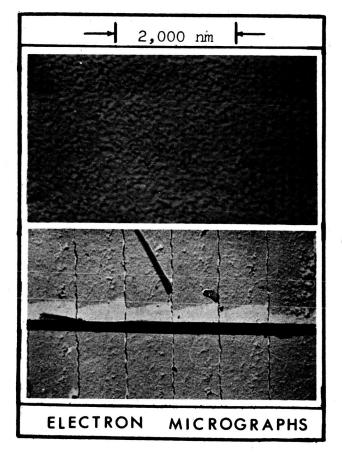
Difficult to burnish

Burnishing - Complete, poor

Groove edge wave - .1 groove







OBSERVATIONS

Substrate - Polished plate
Deposition - With NRL 47
50 microtorr
Electron gun, C crucible
Au: 400 nm: 4.5 nm/sec

Ruling - 1280/mm

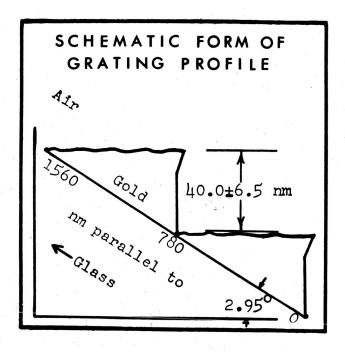
.3 fringe burr

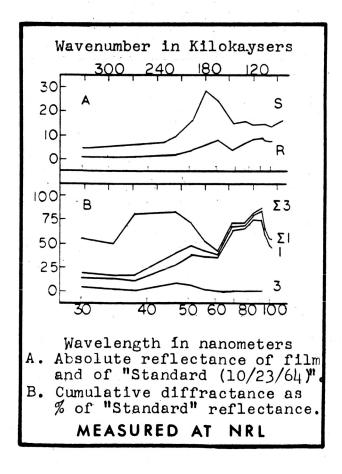
Diamond not bothered

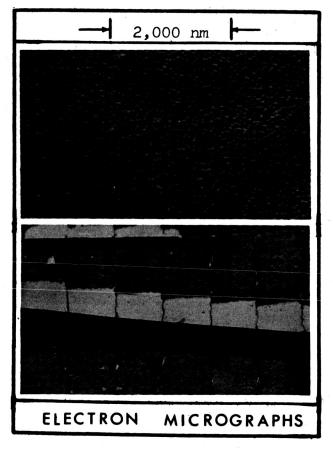
by nuggets

Good ruling Burnishing - Complete

Groove edge wave - .1 groove

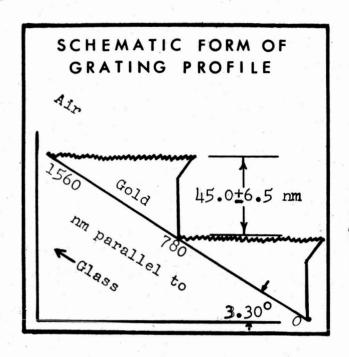


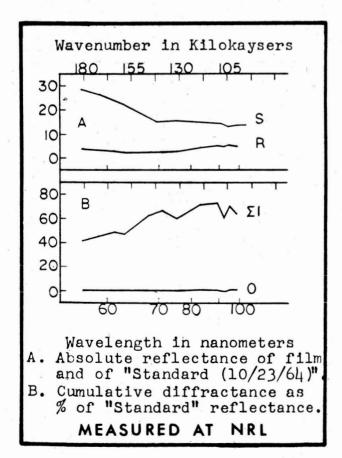


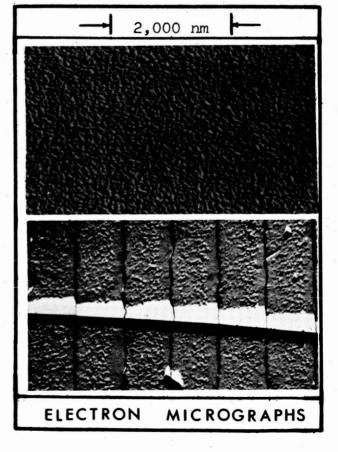


OBSERVATIONS

Substrate - Under-liquid polish
Deposition - With NRL 46
50 microtorr
Electron gun, C crucible
Au: 400 nm: 4.5 nm/sec
Ruling - 1280/mm
.2 fringe burr
Au adhesion to tool
Groove shape good
Rough surface of grooves
Burnishing - Complete, rough
Groove edge wave - .2 groove







OBSERVATIONS

Substrate - Polished glass

Deposition - 12 microtorr

Joule heat, Mo boat

Au: 440 nm: 22 nm/sec

Ruling - 1280/mm

.3 fringe burr

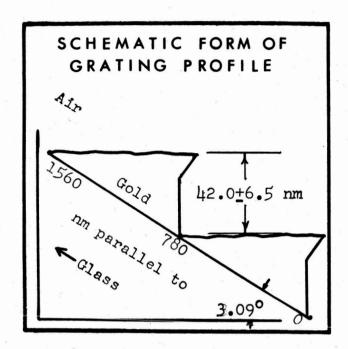
Streaks due to complete

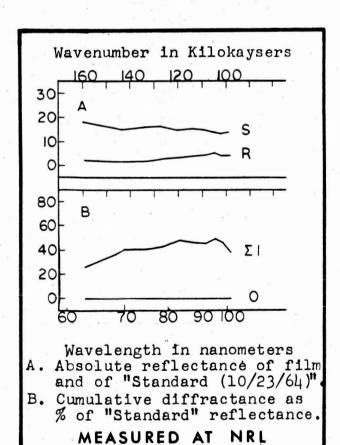
evaporation from boat

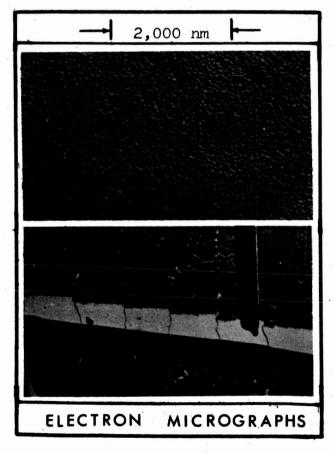
Poor groove form

Burnishing - Complete

Groove edge wave - .4 groove







OBSERVATIONS

Substrate - Polished glass
Deposition ll microtorr
Joule heat, Mo boat

Au: 130 nm: 8.5 nm/sec

Ruling - 1280/mm

.3 fringe burr

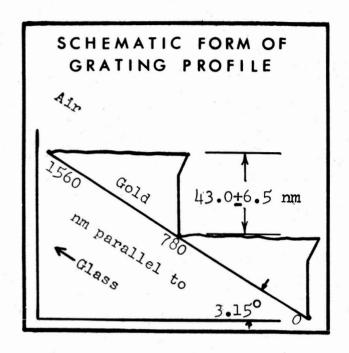
Serious gold adhesion

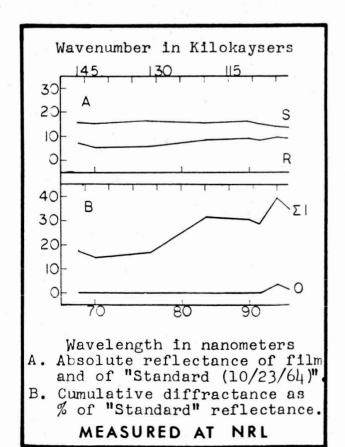
after early grooves

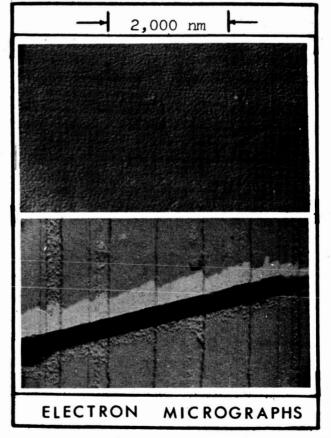
Poor overall

Burnishing - Complete

Groove edge wave - .1 groove







OBSERVATIONS

Substrate - Polished glass

Deposition - 9.5 microtorr

Joule heat, Mo boat

Au: 270 nm: 13.5 nm/sec

Ruling - 1280/mm

.3 fringe burr

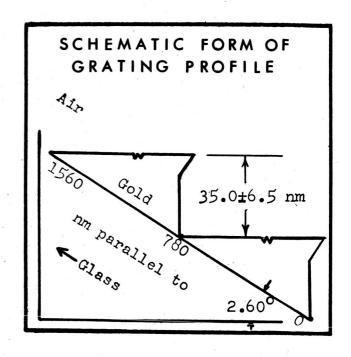
Serious gold adhesion,

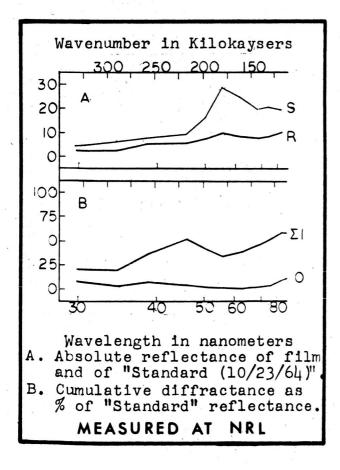
some spots like 48

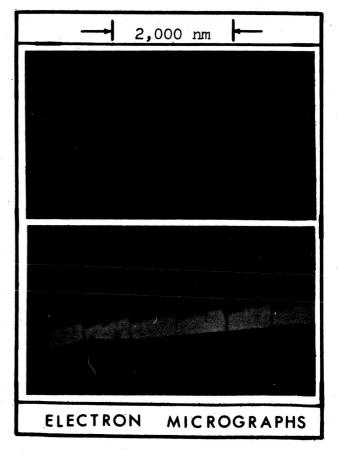
Did not rule well

Burnishing - .9 complete

Groove edge wave - .2 groove







OBSERVATIONS

Substrate - Shined plate

Deposition - With NRL 52

30 microtorr

Joule heat, Mo boat

Au: 160 nm: 16 nm/sec

Ruling - 1280/mm

.25 fringe burr

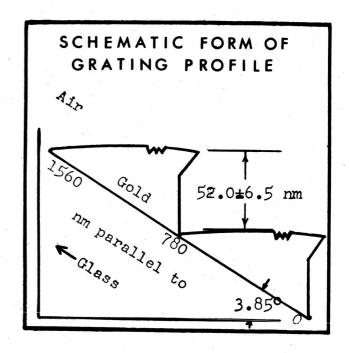
Some Au adhesion and

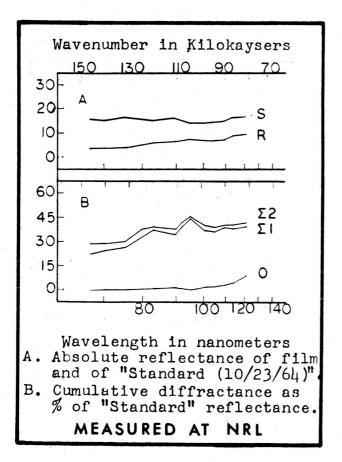
streaks from it

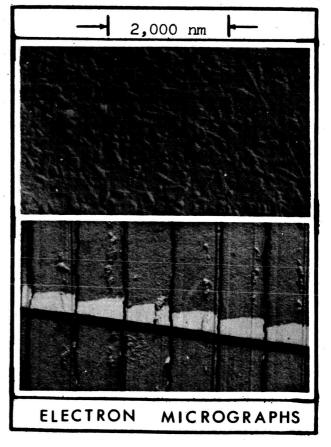
Good ruling

Burnishing - Complete

Groove edge wave - 2 groove







OBSERVATIONS

Substrate - Polished glass
Deposition - With NRL 51
30 microtorr
Joule heat, Mo boat
Au: 160 nm: 16 nm/sec
Ruling - 1280/mm
.3 fringe burr
Some heavy streaks due
to Au adhesion
Similar to 51

Groove edge wave - .1 fringe

Burnishing - Complete

